Specifications for a 200KV Scanning/Transmission Electron Microscope (S/TEM)

GENERAL

Procurement for a high resolution analytical Scanning Transmission Electron Microscope (S/TEM) with x-ray energy dispersive spectroscopy (EDS), a High Angle Annular Dark Field Detector (HAADF), and Electron Energy Loss spectroscopy (EELS) including the following items:

ITEM 1. (Required) The Contractor shall provide a Scanning/Transmission Electron Microscope (S/TEM) which shall have the following features and capabilities:

Item 1A. Vacuum System

1. Specimen area, electron source chamber, and microscope column down to the final projector lens must be pumped by an oil-free pump. Vacuum surfaces and components in the vicinity of the electron beam shall not be subject to degassing or degradation under electron or x-ray radiation.

2. A liquid nitrogen cold trap around the specimen and a liquid nitrogen Dewar shall be included. Cold trap shall be compatible with detectors and specimen holders as specified. A cryo-cycle automatic routine for clean column and vacuum system will be provided.

3. A column vacuum of less than 10^{-6} Pa and FEG gun vacuum of less than 10^{-7}Pa must be maintained, with partial pressures of hydrocarbons less than 4×10^{-8} Pa and of water vapor less than 8×10^{-6} Pa.

4. The vacuum system shall be completely fail safe.

Item 1B. Specimen Airlock / Stage

1. Specimen holder insertion shall be through an automatic pre-pumped airlock. Specimen exchange shall be possible with high voltage and filament heating engaged.

2. It is highly desirable that the airlock and stage shall be compatible with government owned specimen holders that were used on a Phillips CM 200. Inventory includes; two single tilt, one low background single tilt, two low background double tilt, one

single tilt liquid nitrogen, one single tilt heating, one low background tilt/rotate, and one single tilt multiple specimen holder.

3. Contractor shall supply a low background single tilt holder and a low background double tilt holder, both with mechanisms capable of holding brittle specimens without damage. A safe entry key directing the insertion into the right lens is preferred. The sample height in the holder should be adjustable to >0.7 mm to get a larger horizontal field of view for EDS and to accommodate a variety of sample choices.

4. TEM stage should be stable with a maximum allowable drift of 0.5nm nanometer per minute.

5. Specimen stage shall be a computerized, 5-axes eucentric, goniometer (X, Y, Z, alpha, beta), side entry design with stage tilt range of ± 90 degrees for zone-axis alignment and tomography. All five axes shall be computer controlled. Minimum specimen tilt angle (α/β) should be ± 30degree. Maximum tilt for tomography application should be ± 75 degrees or more.

6. The system shall display X, Y, and Z to at least 0.1 μm and alpha and beta angles to at least 0.1 degree and provide storage and recall of a minimum of 20 sets of stage positions as defined by the five axes.

7. X and Y movements shall maintain vertical and horizontal position on the viewing screen at all magnifications. The relative speed of image movement shall be selectable and remain constant at all magnifications.

8. The specimen stage shall have a direct position measurement system that is free of backlash, orthogonal hysteresis, and run-on.

9. X/Y movement range shall be 2mm or more. Fine X and Y adjustments shall allow specimen movements as small as 2 nanometers. Reproducibility and absolute measurement of X/Y positioning shall be less than 0.5 μm and angular positioning shall be less than 0.01 degree.

10. The control software shall include an alpha wobbler for fine adjustment of Z to the eucentric position.

Item 1C. <u>Electron Illumination Source</u>

1. Range of accelerating voltages shall be to a maximum of 200KV with continuous control in selectable step sizes.

2. A factory aligned Field Emission Gun (FEG) with high stability, brightness, and temporal coherence must be included. It should have its own vacuum system. Available probe current at 200KV should be ≥ 1 nA for a 1 nm probe.

3. The maximum beam current should be >150nA at 200KV

4. All column alignments shall be carried out using electromagnetic beam deflectors.

Item 1D. <u>Illumination System</u>

1. The condenser lens aperture holder shall be capable of accepting four (4) standard or low background 3 mm apertures which shall be motorized and computer controlled.

2. Contractor shall include three (3) sets of four (4) low background apertures of standard sizes.

3. The illumination system shall be controlled such that 1) the intensity remains constant with changing magnification, or 2) the maximum intensity is limited, or 3) allows for unrestricted control.

4. Illumination system shall be constructed to minimize generation of spurious x-rays.

Item 1E. <u>Objective Lens</u>

1. Objective lens should be ultra-stable twin lens with constant power. Its design shall allow the specimen to remain at the eucentric position for all operating modes and be able to integrate the ESD system.

2. Objective lens shall have a Scherzer resolution (point resolution) of less than or equal to 0.3 nm, and information limit of less than or equal to 0.12 nm at 200 KV.

3. Electrical stability shall be less than one (1) part per million per minute at 200 KV.

4. The system shall automatically adjust focus step size with magnification with manual override possible.

5. Objective lens shall be at constant excitation to at least 600,000X with minimal thermal changes in the specimen area and minimal adjustment of the objective stigmator.

6. Objective aperture mechanism shall hold a minimum of four different size apertures and should be fully computer controlled.

7. Contractor shall include three (3) sets of four (4) platinum apertures of standard sizes.

Item 1F. Imaging System

1. The imaging system will have a high speed digital camera capable of handling all alignments and applications.

2. In addition, the scope must be equipped with a fast (at least 25fps @ 512 X 512) 4K X 4K 16M (pixel) camera that is usable at 20-200KV HT range, has a large field of view and is capable of providing drift free imaging and precise sample navigation. TEM maximum magnification achievable on the TEM camera plane should be at least 1,500,000X at 200KV.

3. The imaging system and camera should be capable of rotation free high resolution imaging and in-situ dynamic observations with at least 16-bit dynamic range. The camera should be suitable for diffraction and low dose application. It should be retractable when required..

4. The whole imaging system should have an enhanced environmental immunity with a minimum effect from air flows and fine room temperature variations to provide stable images.

Item 1G. Diffraction

1. Selected area aperture holder in the diffraction lens shall accept a minimum of four standard 3 mm apertures which should be fully computer controlled.

2. The minimum spot size should be 0.3nm or smaller.

3. The camera length range in the SADP mode should be (14-5700) mm.

4. Maximum convergence angle should be greater than 100 mrad, and the maximum diffraction angle should be ≥ 26 degree.

5. Dark field imaging shall be performed by tilting the beam using either X-Y or conical controls. Tilt parameters shall be displayed on the main control screen.

6. Software shall provide a calibrated shift capability to determine lattice spacing and crystal structures directly in the microscope.

7. STEM shall be able to form convergent beam diffraction patterns of areas down to < 2 nm diameter with the specimen in the eucentric position. Convergence angle shall be continuously variable to make the diffraction discs as large as possible without overlapping.

Item 1H. <u>Scanning System</u>

1. A complete STEM system unit (with on axis retractable Bright Field (BF)/Dark Field (DF) and High Angle Annular dark Field (HAADF) detectors) that is compatible with TEM system will be provided. The HAADF detector should have a resolution of 0.16nm or better.

2. The STEM system should be a high throughput system with 4 parallel imaging channels available to acquire four or more images at the same time from different STEM detectors. Detectors shall not alter in any way the specification of the basic instrument or specimen tilt range.

3. At least one of the Dark Field (DF) STEM detectors should have 4 segments that can be read individually.

4. STEM should have the availability of a 3-fold condenser astigmatism.

5. Drift Corrected Image Integration (DCFI) option should be available.

6. Maximum STEM magnification should be at least 200Mx.

7. STEM Tomography should be automated with dynamic focusing capability.

Item 1I. <u>X-ray Energy Dispersive Spectroscopy (EDS) Provisions</u>

1. Design of the STEM shall provide for an obstruction-free EDS detector, which should incorporate at least 2 Silicon Drift Detectors (SDD) with a windowless design for light element sensitivity. EDS microanalysis should be fully integrated into the microscope's user interface.

2. The EDS solid angle should be 0.45 steradians (srad) or more.

3. The pixel dwell times should be $\leq$ 10 μsec for automatic collection of element maps and auto-drift correction during acquisition. The detector should be able to provide high precision EDS quantitative composition analysis with 512x512 pixel map in < 30sec.

4. Software shall provide for an x-ray detector protection function.

5. X-ray detector shall have an energy resolution equal to or better than 136 eV at manganese K alpha and 10 Kcps output. X-ray detector shall be capable of detecting boron.

6. EDS maximum output count rate should be 140,000Counts/sec or better.

7. EDS 3D tomography should be available with tilt range of ± 75 deg.

8. An anti-contamination device (LN2) should be provided with the time without refill of 4 four days or longer.

9. For a typical analysis, the EDS peak to background ratio should be > 4000.

10. Application software shall be included for x-ray spectra acquisition and analysis. Full qualitative and quantitative software shall be included with ZAF, Phi Rho Z, and thin film matrix correction algorithms.

Item 1J. <u>Computer and Software</u>

1. Both microscope control (stage position and column settings), all image acquisition, and EDS functions shall be performed by an integrated software system.

2. The support PC and the monitors (two monitors with at least 27" diagonal measurement) shall be provided.

3. The microscope software platform shall be based on the 64-bit Microsoft Windows 7 operating system. It shall be user friendly and capable of fast acquisition and analysis of TEM, STEM, and EDS data.

4. The software shall allow for the automation of the acquisition and the analysis of TEM, STEM, and EDS data. It will also allow for the storage and recall of lens settings and stage positions.

5. Software shall control the stage to perform automatic grid searches, image montages, storage and recall of stage's five axes' positions.

6. The software shall provide a facility to accurately measure distances, heights, ratios, and angles for all scanned images and diffraction patterns.

7. The software shall be capable of performing drift corrected frame integration where frames are automatically aligned over a user selectable number of frames.

8. Application software shall be included for electron pattern interpretation and analysis.

9. Hardware and software for STEM interface and beam control shall be included. Digital image fast and slow scan collection, display, image enhancement, image archiving, x-ray dot mapping, image analysis and measurement shall be included.

10. To maximize operator efficiency and to minimize operator interference, full remote operation of the scope shall be possible.

Item 1K. <u>Installation and Training</u>

1. Contractor shall furnish all labor, material, tools, transportation, and lodging for installation and will provide a list of installation site requirements.

2. Contractor shall provide a mains matching transformer capable of inputs from 190 to 440 volts and output of 220 volts. Transformer shall include proper isolation and filtering to prevent interference from accessories.

3. Contractor shall supply an air compressor to meet the STEM's requirements.

4. Contractor shall provide a radiation alarm monitoring system and sulfur hexafluoride detector which shall be approved by the on-site safety office.

5. Contractor shall provide a water cooled water chiller.

6. Contractor shall provide a warranty of a minimum of twelve months.

7. Contractor shall provide operation and software manuals including electrical and mechanical drawings.

8. Contractor shall provide training (a minimum of sixteen (16) hours of on-site training) and demonstrate required performance.

9. It is highly desirable that the contractor directly provide service for all system components: TEM, all detectors, associated electronics and software, pumps, and chiller. This would preclude the need for multiple service contracts/contacts for subsequent system maintenance.

10. It is highly desirable that the contractor provide a trade-in discount based on the value of GRC’s current TEM: a Phillips CM-200. This would also include removal costs.

<u>Item 2</u>. (Optional) The contractor shall provide an Electron Energy Loss spectroscopy (EELS) system which shall have the following features and capabilities;

Item 2A. <u>Design</u>

1. The post-column EELS spectrometer shall be compatible with item 1 and shall be capable of operating up to and including 200 KV.

2. Maximum acquisition rate in spectroscopy shall be 1000spectra/sec or better. Maximum collection angle for EELS shall be 200mrad or better.

3. The aperture size should be 3mm, energy resolution (FWHM) of 0.40 eV and energy field of view 1024 eV.

4. All the interface housing, flanges etc. required to mount the EELS spectrometer will be provided.

Item 2B. <u>Computer and Applications Software</u>

1. Application software shall be included for image acquisition and analysis.

2. Application software shall be included for energy loss spectrum acquisition and quantification. Elemental mapping of at least 512 x 512 pixels shall be included. Software shall be capable of standard-less quantification using built in cross-sections for all elements.

Item 2C. <u>Installation and Training</u>

1. Contractor shall provide all labor, material, tools, transportation, and lodging for installation.

2. Installation shall include alignment at 200 KV and an additional voltage.

3. A minimum of sixteen (16) hours of on-site training shall be included.

4. Contractor shall provide a warranty of a minimum of twelve (12) months.

5. Contractor shall provide operation and software manuals including electrical and mechanical drawings.

6. Contractor shall provide alignment maintenance for a period of two (2) years.

Anita Garg, x8908, MS 49-3
Pete Bonacuse, x3309, MS 49-3